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**0708482.5**

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**Application number GB**

**UK - IPO  
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**- 2 MAY 2007**

**NEWPORT**

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1. Your reference: **P101097.GB.01/NHE**  
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**8333072003**

3. Title of the invention: **Biomass Treatment Process**

4. Name of your agent (if you have one): **Murgitroyd & Company**

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Date: 01/05/2007

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## BIOMASS TREATMENT PROCESS

The present invention relates to the processing of biomass for the purpose of producing biofuels. More specifically, the invention is a process for pre-treating biomass in order to aid the biofuel production process. The invention is particularly suited, although not exclusively so, to the pre-treatment of lignocellulosic biomass for use in ethanol production.

The conversion of biomass into biofuel has taken on great significance in recent years as consumers and producers alike recognise the environmental and sustainability issues surrounding existing fossil fuels. The bulk of existing biofuel is derived from the fermentation of sugar crops and the like, but these crops have a high value in food applications and their sugar yield per hectare is low when compared to cellulose and hemicellulose. Thus, the derivation of biofuel from alternative sources of biomass, such as lignocellulosic biomass composed primarily of lignin and cellulose, is of great significance to producers.

Lignocellulosic biomass is an extremely abundant biomass. It includes all trees and grasses, as well as agricultural residues such as wet and dry distiller's grains, straw and sugarcane bagasse. Lignocellulosic biomass can be converted into ethanol by hydrolysis and subsequent fermentation. In hydrolysis, the cellulosic part of the biomass is converted to sugars, and the fermentation converts these sugars to ethanol. To increase the yield of the hydrolysis, a pre-treatment step is needed to soften the biomass and disrupt its cellular structure.

Disruptive pre-treatment processes are normally chemical or physical in nature. Current chemical pre-treatment processes rely on a catalyst to achieve the desired disruption of the cells of the biomass. This catalyst is

commonly an acid or an enzyme. The acid has the disadvantage of being harmful to the environment, whilst enzymes are relatively expensive. The most common physical pre-treatment process is steam explosion, examples of which are disclosed in US4425433 and US4461648. In  
5 steam explosion, the biomass is heated using high pressure steam for a few minutes, before the reactions are stopped by a sudden decompression to atmospheric pressure. A disadvantage of steam explosion is that the process must be contained within a suitable process vessel, and is thus a non-continuous process. Furthermore, the sugar  
10 yields from steam explosion are comparatively low while current costs for the process are high.

It is an aim of the present invention to obviate or mitigate one or more of the aforementioned disadvantages present in existing pre-treatment  
15 processes.

According to the present invention, there is provided a process for the treatment of biomass, comprising:

- 20 forming a biomass slurry by mixing biomass with a working fluid;
- inducing the biomass slurry to flow through an inlet into a passage;
- injecting a high velocity transport fluid into the slurry through a nozzle communicating with the passage;
- applying a shear force to the slurry such that the working fluid is atomised and forms a vapour and droplet flow regime;
- 25 forming an at least partial vacuum within the passage downstream of the nozzle;
- generating a condensation shock wave within the passage downstream of the nozzle and vacuum by condensation of the transport fluid; and

inducing the slurry to flow from the inlet through the vacuum and condensation shock wave.

Preferably, the transport fluid is steam.

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Preferably, the transport fluid is injected at a supersonic velocity.

Preferably, the passage is of substantially constant diameter. Preferably the nozzle is an annular nozzle which circumscribes the passage.

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Preferably, the process is a batch process in which the processed slurry is returned to the inlet. Alternatively, the process is an in-line process in which the inlet receives slurry from an upstream portion of a biofuel production line, and the passage passes the processed slurry to a downstream portion of the production line.

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Preferably, the process further comprises the step of injecting a catalyst into the slurry as it passes through the vacuum. Alternatively, the process further comprises the step of injecting first and/or second catalysts into the slurry as it passes through the vacuum. The first and second catalysts may be injected simultaneously or consecutively. Where the process is a batch process and the catalysts are to be injected consecutively, the first catalyst is injected into the slurry for a first period of time, and the second catalyst is injected into the slurry for a second period of time occurring after the first period has elapsed.

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Preferably the first catalyst is an acid. Preferably the second catalyst is an enzyme.

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Preferably, the biomass is lignocellulosic biomass.



A preferred embodiment of the present invention will now be described, by way of example only, with reference to the accompanying drawings, in which:

5           Figure 1 shows a cross-section through an apparatus suitable for use in the process of the present invention; and

          Figure 2 shows a graph showing pressure and temperature profiles of a biomass slurry during the process.

10          Referring to Figure 1 there is shown a processing apparatus 1 comprising a housing 2 defining a passage 3 providing an inlet 4 and an outlet 5. Preferably, the passage 3 is of substantially constant circular cross section. The inlet 4 is formed at the front end of a protrusion 6 extending into the housing 2 and defining exteriorly thereof a plenum 8 for the  
15          introduction of a transport fluid, the plenum 8 being provided with an inlet 10. The protrusion 6 defines internally thereof part of the passage 3. The distal end 12 of the protrusion 6 remote from the inlet 4 is tapered on its relatively outer surface at 14 and defines a nozzle 16 between it and a correspondingly tapered part 18 of the inner wall of the housing 2. The  
20          nozzle 16 is in flow communication with the plenum 8 and is preferably annular. The nozzle 16 has convergent-divergent internal geometry in order to accelerate the transport fluid to a high velocity.

          Whether the process is a batch or in-line process, the inlet 4 receives a  
25          biomass slurry which passes out of the outlet 5. Introduction of the transport fluid, which in this non limiting example is steam, into the apparatus 1 through the inlet 10 and plenum 8 causes a jet of steam to issue forth through the nozzle 16. As the steam is injected into the slurry, a momentum and mass transfer occurs between the two which results in  
30          the atomisation of the working fluid component of the slurry. This transfer

is enhanced through turbulence. The steam applies a shearing force to the slurry which not only atomises the working fluid component but also disrupts the cellular structure of the lignocellulosic biomass suspended in the slurry.

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The effects of the process on the temperature and pressure of the slurry can be seen in the graph of Figure 2, which shows the profile of the temperature and pressure as the slurry passes through various points in the processing apparatus of Figure 1. The graph has been divided into four sections A-D, which correspond to various sections of the processing apparatus shown in Figure 1. Section A corresponds to the section of the passage 3 between the inlet 4 and the nozzle 16. Section B corresponds to the upstream section of the mixing chamber 3A extending between the nozzle 16 and an intermediate portion of the chamber 3A. Section C corresponds to a downstream section of the mixing chamber 3A extending between the aforementioned intermediate portion of the chamber 3A and the outlet 5, while section D illustrates the temperature and pressure of the slurry as it passes through the outlet 5.

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The steam is injected into the slurry at the beginning of section B of the Figure 2 graph. The speed of the steam, which is preferably injected at a supersonic velocity, and its expansion upon exiting the nozzle 16 cause an immediate pressure reduction. At a point determined by the steam and geometric conditions, and the rate of heat and mass transfer, the steam begins to condense, further reducing the pressure and causing an increase in temperature. The steam condensation continues and forms a condensation shock wave in the downstream section of the mixing chamber 3A. The forming of a condensation shock wave causes a rapid increase in pressure, as can be seen in section C of Figure 2. Section C

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also shows that the temperature of the slurry also continues to rise through the condensation of the steam.

As explained above, as the steam is injected into the slurry through nozzle  
5 16 a pressure reduction occurs in the upstream section of the mixing chamber 3A. This reduction in pressure forms an at least partial vacuum in this upstream section of the chamber 3A. Tests have revealed that an approximately 90% vacuum can be achieved in the chamber 3A as the steam is injected and subsequently condenses.

10 As previously stated, the shear force applied to the slurry and the subsequent turbulent flow created by the injected steam disrupts the cellular structure of the biomass suspended in the slurry. As the slurry passes through the partial vacuum and condensation shock wave formed  
15 in the chamber 3A, it is further disrupted by the changes in pressure occurring, as illustrated by the pressure profile in sections B and C of Figure 2.

Although not shown in Figure 1, one or more additional nozzles can  
20 optionally be provided immediately downstream of the transport fluid nozzle 16. These additional nozzles would open into the upstream section of the mixing chamber 3A where the vacuum is formed during the processing operation. A single additional nozzle can be connected to a supply of one or more catalysts which can be injected into the slurry to  
25 further disrupt the cellular structure of the biomass. In the instance where the process is a batch process, the injection of the catalyst(s) into the partial vacuum can be timed for a specific period to achieve a specific dosage. The catalysts are preferably acids and enzymes. Where a single additional nozzle is used, it would be connected to separate sources of  
30 acid and enzyme and can inject either or both.

Alternatively, a pair of additional nozzles can be used to inject the catalysts into the partial vacuum, either simultaneously or consecutively. In the instance where the process forms part of a biofuel production line,  
5 the pair of nozzles will inject the catalysts simultaneously.

The additional nozzle(s) may also inject an enzyme only, with no acids being used.

10 Trials of the process of the present invention have been undertaken using lignocellulosic biomass in the form of dried distiller's grains (DDG) and wet distiller's grains (WDG). Using established HPLC analysis, it was found that applying the inventive process to a slurry of DDG converted 21% of the solids present into sugars. In the trial using a slurry of WDG, the same  
15 HPLC analysis established that the present process was successful in converting 17% of the solids to sugars. Further trials have been performed using a fibrous material produced from maize. These trials concluded that the present process was successful in converting 30% of the solid content to sugars, which represents nearly 100% of the available  
20 sugar content.

During a further trial processing sugarcane bagasse there was evidence of wall swelling in the cellular structure of the biomass. This is an indication that the crystalline structure of the cellulose was disrupted during the  
25 inventive process. This swelling of the cellulose facilitates the action of cellulose enzymes and therefore accelerates the process and/or increases the yield of fermentable sugars. This effect could be interpreted as a swelling resulting from scission of hydrogen bonds as a result of the shear forces and mechanical shock caused by the process.

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By injecting steam into the biomass slurry in such a way as to atomise the working fluid, and create a partial vacuum and condensation shock wave through which the slurry passes, the present invention ensures a greater degree of disruption to the cellular structure of the biomass than that  
5 achieved by existing pre-treatment processes. Furthermore, as the disruption is at least partially achieved by the steam injection, the invention enables a reduced amount of catalyst, whether acid or enzyme, to obtain the desired degree of disruption when compared with existing chemical pre-treatment processes. In fact, the disruption achieved by the steam  
10 injection can remove the need for one or more catalysts entirely. The steam injection of the present process ensures continual shearing of the slurry and maintains the partial vacuum and shock wave. The process of the present invention is therefore continuous, with no need to contain the process in a stand-alone vessel such as that required in steam explosion  
15 processes.

Similarly, injecting the catalysts into the partial vacuum created in the process allows the catalysts a higher level of penetration into the biomass. Thus, either a lesser amount of catalyst can be used, or else the same  
20 amount of catalyst can be used as in the existing processes with an improved degree of disruption.

The high shear forces imparted by the high velocity steam injection not only assist in the disruption of the cellular structure of the biomass, but  
25 also atomise the working fluid component of the slurry to ensure intimate and homogenous heating and mixing of the biomass slurry with the catalysts. Such improved heating and mixing reduces the amount of time and catalyst required to achieve the necessary chemical reaction.

The process of the present invention may be used in areas of biofuel production other than those referred to above. One example of another treatment to which the present invention is also suited is maize liquefaction, in which starch is extracted from maize and broken down into short chains of sugars using a catalyst. One existing liquefaction process involves adding a catalyst to a starch slurry, and heating the mixture to a desired temperature (e.g. 85°C) using a jet cooker. The mixture is then held in a vat at the desired temperature for a predetermined period of time while the catalyst reacts with the slurry.

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This heating ensures that the catalyst operates in its optimum temperature range. However, conventional jet cookers inject steam at high temperatures into a product, resulting in the product close to the injection point being heated to a very high temperature whilst product remote from the injection point is much lower in temperature. Catalysts present in the product close to the injection point can therefore be damaged by the very high temperatures. Although mechanical stirrers mix the product and catalysts in the vat, there are still significant temperature differences between the heat-jacketed vat walls and the majority of the product. It is also difficult to achieve a homogenous mix when using conventional mechanical stirrers.

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In using the process of the present invention for such a liquefaction procedure, the starch slurry is passed through the apparatus in the same manner as described above with reference to lignocellulosic biomass, with steam injected at high velocity into the slurry. The intensity of the steam would be adjusted once the desired temperature was reached, such that the slurry was then held at the desired temperature for a predetermined amount of time.

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As during the processing of the biomass slurry, the shear forces of the steam on the starch slurry physically disrupt the cellular structure of the starch, creating a greater surface area for the catalyst to penetrate. The vacuum and condensation shock wave created by the steam injection also assist in the disruption of the cells of the starch. Furthermore, injecting the catalyst via an additional nozzle downstream of the steam nozzle into the vacuum zone reduces temperature damage to the catalyst. Using the process of the present invention for this liquefaction also ensures a more homogenous heating and mixing of slurry and catalyst than is possible with the existing process.

The liquefaction process can be a batch process or an in-line process, which have both already been described above.

The process of the present invention provides the same advantages when used in liquefaction to process starch slurry as it does when used in hydrolysatation of biomass slurry. In other words, the process reduces the amount of enzymes required and also the time taken to achieve the desired degree of liquefaction.

Whilst steam is the preferred transport fluid used in the process of the present invention, it should be understood that other suitable fluids may also be used instead.

The additional nozzles referred to above can also include a control means which monitors the flow rate of the slurry through the passage and meters the dosage of catalyst introduced by the nozzle(s).

In the in-line embodiment of the present invention, a number of units of the aforementioned apparatus may be placed in series. In this instance, units

can be used to heat the slurry more rapidly than a single unit.

Alternatively, the multiple units can be used for differing purposes. For example, a first unit can apply the process of the present invention and inject a chemical pre-treatment catalyst while a subsequent unit can apply the process again but introduce an enzyme.

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Other modifications and improvements may be incorporated without departing from the scope of the invention.





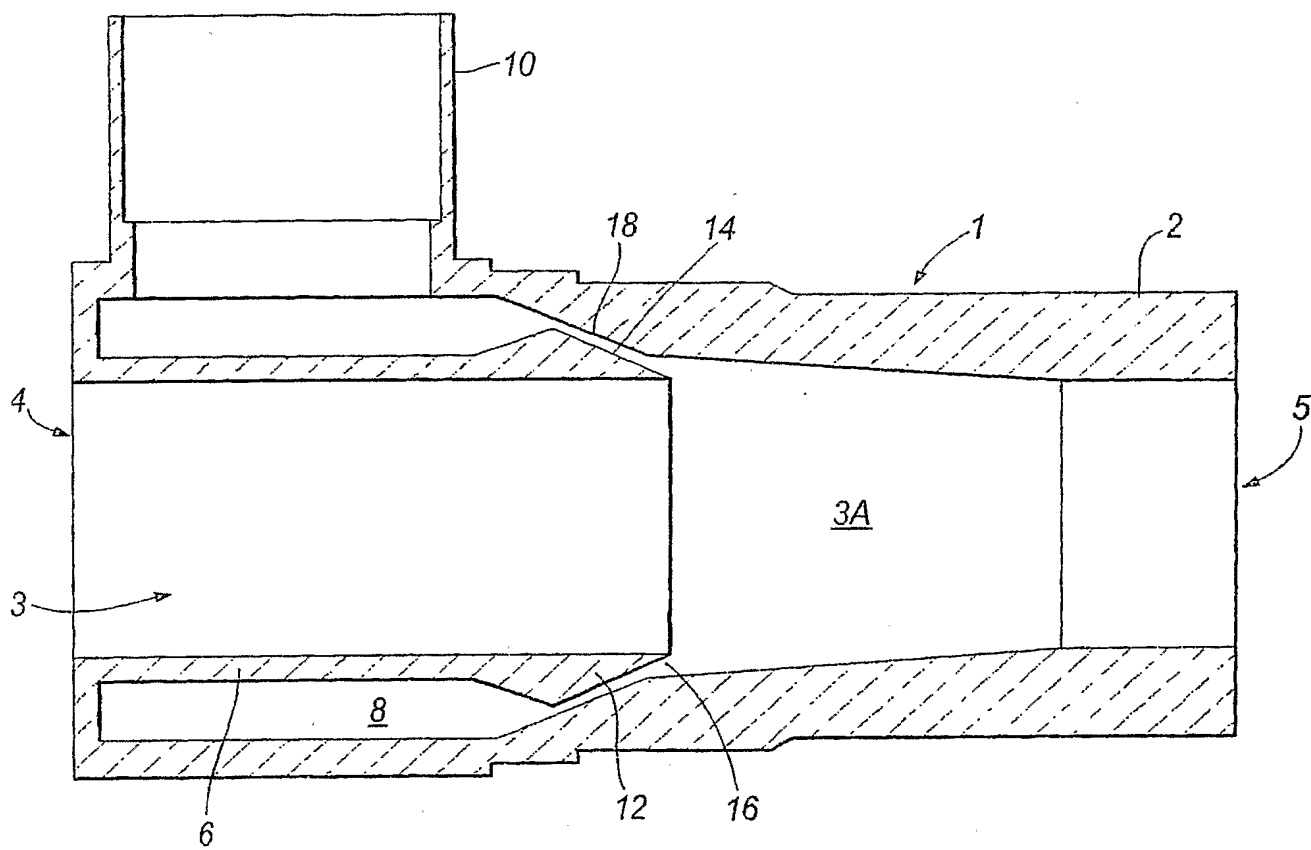
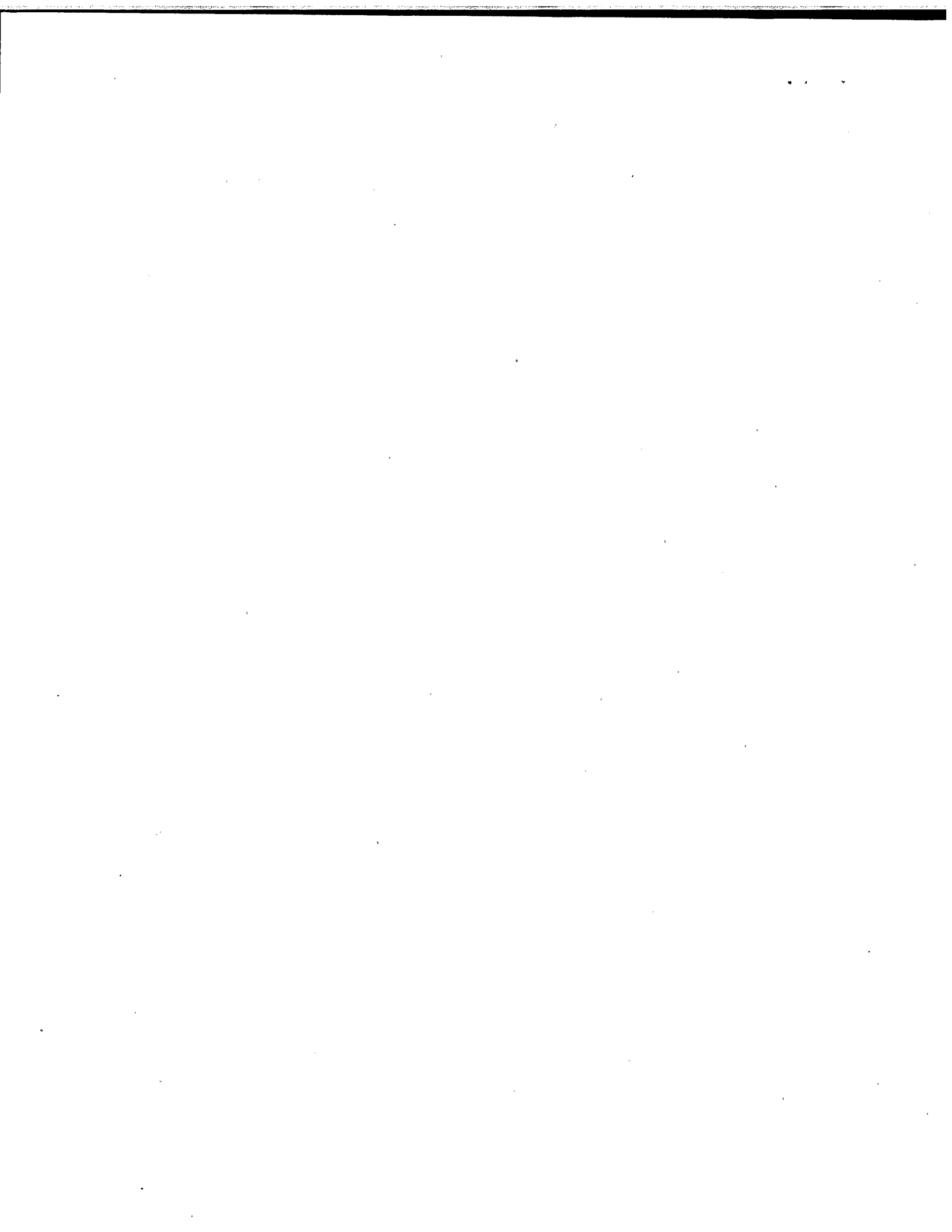


Fig. 1



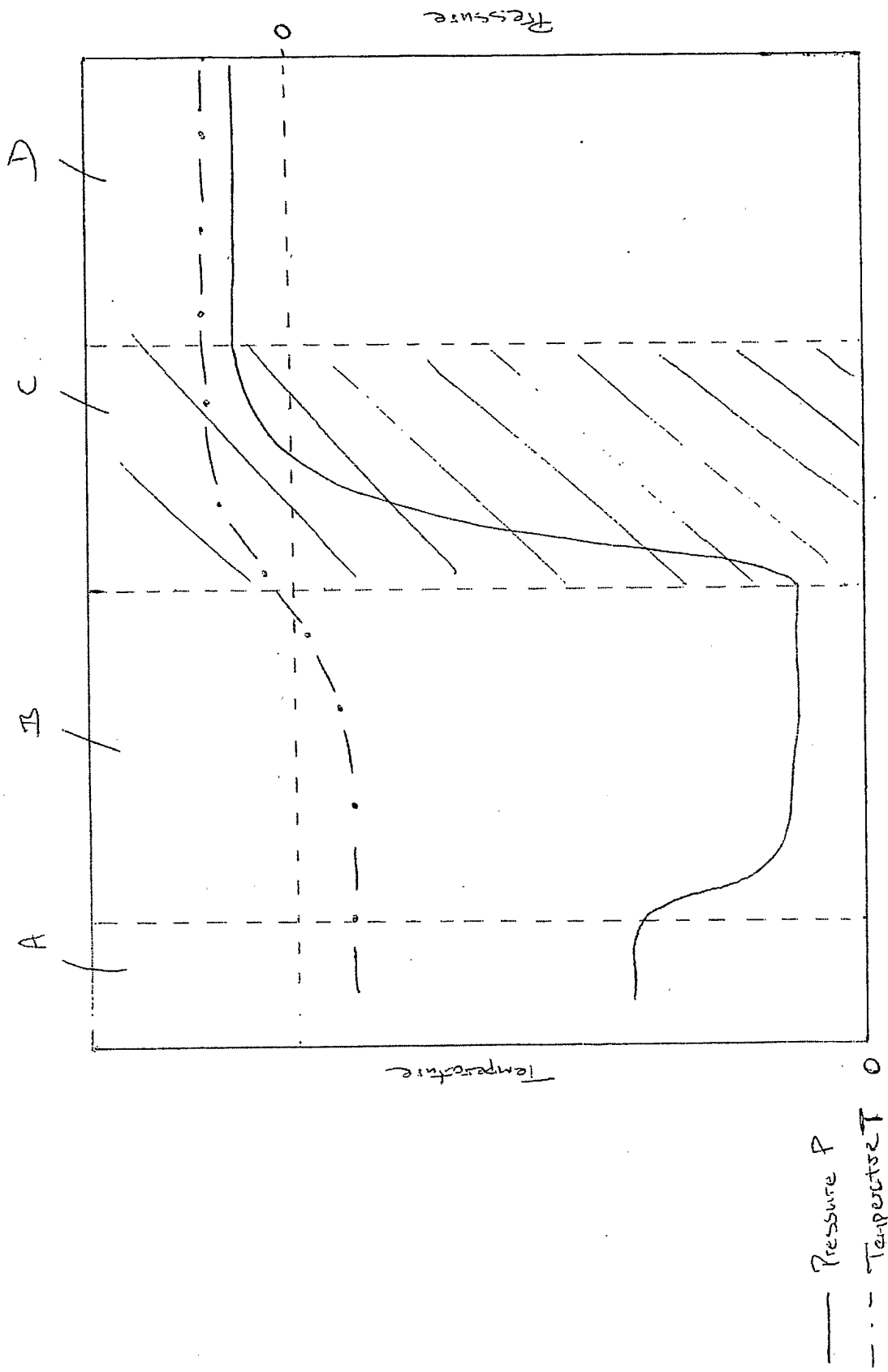


Fig. 2

